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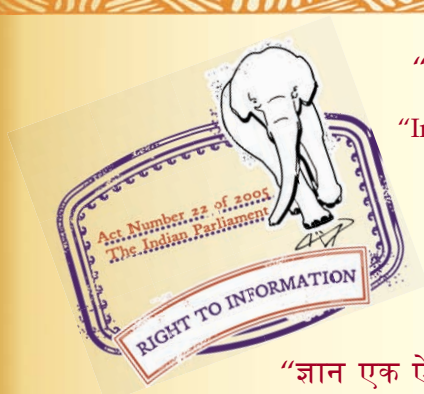
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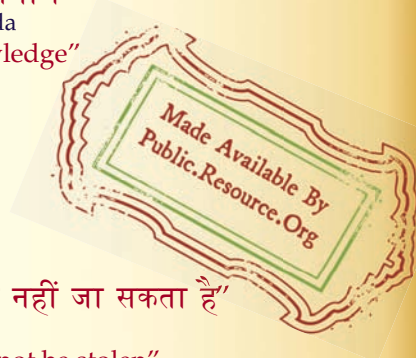
IS 1889-3 (1979): Method for Quantitative Chemical Analysis of Binary Mixtures of Regenerated Cellulose Fibres and Cotton, Part III: Formic Acid-zinc Chloride [TXD 5: Chemical Methods of Test]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

METHOD FOR
QUANTITATIVE CHEMICAL ANALYSIS OF
BINARY MIXTURES OF REGENERATED
CELLULOSE FIBRES AND COTTON
PART III FORMIC ACID-ZINC CHLORIDE

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METHOD FOR QUANTITATIVE CHEMICAL ANALYSIS OF BINARY MIXTURES OF REGENERATED CELLULOSE FIBRES AND COTTON

PART III FORMIC ACID-ZINC CHLORIDE

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SHRI M. D. DIXIT

DR A. S. DUTT

Office of the Textile Commissioner, Bombay

The Bombay Textile Research Association, Bombay
Indian Jute Industries' Research Association,
Calcutta

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Textiles Committee, Bombay

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Indian Standard

METHOD FOR QUANTITATIVE CHEMICAL ANALYSIS OF BINARY MIXTURES OF REGENERATED CELLULOSE FIBRES AND COTTON

PART III FORMIC ACID-ZINC CHLORIDE

0. FOREWORD

0.1 This Indian Standard (Part III) was adopted by the Indian Standards Institution on 10 January 1979, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

0.2 The Indian Standard on method for quantitative chemical analysis of binary mixtures of regenerated cellulose fibres and cotton, when first published in 1962 covered only sodium zincate method. While taking up the revision of this standard, the Committee decided to cover Cadoxen solvent, formic acid-zinc chloride and sulphuric acid methods also as Parts II, III and IV of this standard respectively.

0.3 The use of different fibre blends in textiles has necessitated the formulation of standard methods for identification and quantitative estimation of respective fibres. The quantitative analysis of textile fibres in mixtures is of considerable importance to textile technologists, traders and consumers.

0.4 In the preparation of this standard (Parts I to IV) due weightage has been given to the test methods prevalent in the industry and trade and also the methods developed by International Organization for Standardization (ISO). Parts I and III are based mainly on ISO 1833-1973 'Textiles—Binary fibre mixtures—Quantitative chemical analysis'. Part II is based on the method developed by Prof W. B. Achwal of the University of Bombay (Department of Chemical Technology), Bombay and the extensive experiments carried out at The Ahmadabad Textile Industry's Research Association, Ahmadabad; The Bombay Textile Research Association, Bombay; and The Silk & Art Silk Mills' Research Association, Bombay. Part IV is based on the work done by Textiles Committee, Bombay and other Textile laboratories.

0.5 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part III) prescribes formic acid-zinc chloride method for quantitative chemical analysis of binary mixtures of regenerated cellulose fibres and cotton in textile form, such as fibre, yarn or fabric.

NOTE — Before conducting an analysis according to this method, the fibres present in the mixture should be identified (see IS : 667-1955†) and the sample to be analysed shall be freed from all non-fibrous matter by the method given in Appendix A. Dye in the dyed fibres is considered to be an integral part of the fibre and is not to be removed.

1.1.1 This method is not applicable to mixtures in which the cotton has suffered extensive chemical degradation, nor when the viscose, cupro or modal fibre is rendered incompletely soluble by the presence of certain permanent finishes or reactive dyes that cannot be removed completely.

2. PRINCIPLE

2.1 A sample of the mixture is dried and weighed. The regenerated cellulose fibres in the sample are dissolved in formic acid-zinc chloride reagent. The residue (cotton) is collected, washed, dried and weighed; and the proportion of regenerated cellulose and cotton is calculated.

3. SAMPLING

3.1 Lot — The quantity of textile material of definite type and quality delivered to one buyer against one despatch note shall constitute a lot.

3.1.1 If the textile material is fibre or yarn and the lot consists of more than 200 kg of the material, it shall be divided into sub-lots weighing 200 kg or less and each sub-lot shall be tested separately.

3.2 Sampling for Fibre and Yarn — From a sub-lot, 15 increments weighing approximately 10 g each shall be taken from different parts and mixed thoroughly to constitute a **test sample**.

3.3 Sampling for Fabrics — From each piece in the gross sample selected as in IS : 5463-1969‡ cut out small portions from at least two different parts weighing about 25 g. Dissect the small portions of the fabric thus collected into yarn and mix thoroughly. This shall constitute a **test sample**.

*Rules for rounding off numerical values (revised).

†Simple methods for identification of common commercial textile fibres.

‡Methods for sampling of cotton fabrics for chemical tests.

4. APPARATUS

4.1 Sintered Glass Filter Crucibles — of appropriate capacity with pore size of 90 to 150 microns (porosity 1) and fitted with ground glass stopper. If the stoppers are not available, the crucibles should be enclosed in weighing bottles for weighing.

4.2 Conical Flask — of 100 ml capacity provided with a glass stopper.

4.3 Analytical Balance — of an accuracy of 0.0002 g.

5. REAGENTS

5.0 Quality of Reagents — Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS:1070-1977*) shall be used where the use of water as a reagent is intended.

NOTE - 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

5.1 Formic Acid-Zinc Chloride Reagent — Dissolve 20 g of anhydrous zinc chloride (analytical reagent quality) and 68 g of anhydrous formic acid in water and make up to 100 g, specific gravity 1.367. If anhydrous formic acid is not available, equivalent quantity of 80 to 85 percent formic acid may be used.

5.2 Dilute Ammonia Solution — 20 ml of concentrated ammonium hydroxide (specific gravity 0.88) made up to one litre with water.

6. PREPARATION OF TEST SPECIMENS

6.1 From each sample (3.2 or 3.3), after removing size and finishes as prescribed in Appendix A, draw a representative specimen weighing about 2 to 3 g. Cut the test specimen into pieces of approximately 25 mm length.

7. PROCEDURE

7.1 Take a test specimen weighing about 0.5 g and put it in a weighing bottle. Dry the specimen in the weighing bottle at $105 \pm 3^\circ\text{C}$ to constant mass and obtain oven-dry mass of the specimen.

7.2 Put the oven-dry specimen without any delay in a 100-ml glass-stoppered conical flask pre-heated to 70°C , and add to it 50 ml of formic acid-zinc chloride reagent also pre-heated to 70°C . Stopper the flask, shake it and keep it in a thermostatically controlled bath at $70 \pm 2^\circ\text{C}$ for 20 minutes. During this period, shake the flask a couple of times. Filter the contents through a tared sintered glass crucible avoiding the transfer of residue.

*Specification for water for general laboratory use (second revision).

7.3 Wash the residue (undissolved cotton) thoroughly with water at 40°C by decantation. Add 100 ml dilute ammonia solution and keep for 10 minutes. Filter through the crucible under suction, wash with water and dry the residue at $105 \pm 3^{\circ}\text{C}$ to constant mass.

8. CALCULATION

8.1 Calculate the percentage of cotton fibre in each test specimen by the following formula and determine the average of all the values:

$$\text{Percentage of cotton} = \frac{100 \times M_r \times d}{M_s}$$

where

M_r = mass of residue obtained in **7.3**,

M_s = mass of specimen obtained in **7.1**, and

d = correction factor for the loss in mass sustained by cotton during the analysis.

NOTE — The value of d is found to be 1.06 for raw cotton and 1.02 for bleached cotton.

9. REPORT

9.1 The report shall include the following information:

- a) Type of the material,
- b) Percentages of:
 - 1) regenerated cellulose,
 - 2) cotton, and
- c) Number of specimens tested.

A P P E N D I X A

(*Note under clause 1.1, and clause 6.1*)

METHOD FOR REMOVAL OF SIZE AND FINISHES FROM TEST SAMPLES

A-1. From each test sample, draw a representative sample weighing about 10 g and dry it at $105 \pm 3^{\circ}\text{C}$ for 3 hours. Treat the sample as given in **A-2** to **A-6**. However, if the nature of the finish present in the sample is known only relevant steps may be carried out.

A-2. Extract the sample with benzene-methyl alcohol mixture in a Soxhlet apparatus for 2 hours at a minimum rate of 6 cycles per hour. (This removes oils, fats, waxes, certain thermoplastic resins, etc.)

A-3. Extract the sample with ethyl alcohol in a Soxhlet apparatus for 2 hours at a minimum rate of 6 cycles per hour. (This removes soaps, cationic finishes, etc.)

A-4. Treat the sample with 200 ml of water at 50°C for 30 minutes, stirring occasionally with glass rod or mechanically. Rinse thrice with fresh portion of warm water (50°C) and dry. (This removes water-soluble materials.)

A-5. Give an enzyme treatment to the sample under conditions depending upon the nature of enzyme used and rinse thoroughly. (This removes starch, gelatine, etc.)

A-6. Immerse the sample in 200 ml of 0.1 N hydrochloric acid at 80°C for 25 minutes, stirring gently every 3 minutes. Rinse thoroughly with water at 80°C containing a few drops of ammonia and then finally with plain water. Remove excess water from the sample by squeezing, suction or centrifuging and allow to dry. (This removes resins.)

INDIAN STANDARDS

ON

CHEMICAL METHODS OF TEST FOR QUANTITATIVE CHEMICAL ANALYSIS OF FIBRE MIXTURES

IS:

- 667-1955 Simple methods for identification of common commercial textile fibres
- 1564-1962 Method for quantitative chemical analysis of binary mixtures of cellulose triacetate and certain other fibres
- 1889 Method for quantitative chemical analysis of binary mixtures of regenerated cellulose fibres and cotton:
 - 1889 (Part I)-1976 Sodium zincate method (*first revision*)
 - 1889 (Part II)-1976 Cadoxen solvent method
- 2005-1962 Method for quantitative chemical analysis of binary mixtures of polyamide fibres and certain other fibres
- 2006-1978 Method for quantitative chemical analysis of binary mixtures of protein fibres and certain other fibres (*first revision*)
- 2176-1962 Method for quantitative chemical analysis of binary mixtures of secondary cellulose acetate and certain other fibres
- 2177-1962 Method for quantitative chemical analysis of mixtures of cellulose triacetate and secondary cellulose acetate fibres
- 2727-1964 Method for quantitative chemical analysis of binary mixtures of manila and sisal fibres
- 3416-1966 Method for quantitative chemical analysis of mixtures of polyester fibres with cotton or regenerated cellulose
- 3421-1966 Method for quantitative chemical analysis of binary mixtures of acrylic and certain other fibres
- 6503-1972 Method for quantitative chemical analysis of ternary mixtures of protein fibres, nylon 6 or nylon 6'6, and certain other fibres
- 6504-1972 Method for quantitative chemical analysis of ternary mixtures of viscose rayon, cotton and protein fibres
- 6570-1972 Method for quantitative chemical analysis of binary mixtures of jute and animal fibres